

VOLUME 34

NOVEMBER 1956

NUMBER 6

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**Published by THE NATIONAL RESEARCH COUNCIL
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(Formerly Section F, Canadian Journal of Research)

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Canadian Journal of Technology

Issued by THE NATIONAL RESEARCH COUNCIL OF CANADA

VOLUME 34

NOVEMBER 1956

NUMBER 6

THE EXTRACTION OF URANIUM FROM A SASKATCHEWAN PEGMATITE GRANITE¹

BY BRAD GUNN, S. D. CAVERS,² AND A. B. VAN CLEAVE

ABSTRACT

Extensive leaching experiments have been done with the object of determining efficient and economic methods for the recovery of uranium from a Saskatchewan pegmatite granite which contains 0.06 to 0.07% of U_3O_8 . Uranium recoveries of 90% or better were obtained by leaching relatively coarsely ground rock (<0.185 in.) for periods of from 48 to 72 hr. at a pulp density of 50% with a solution containing 36 lb. of concentrated sulphuric acid per ton of ore without the addition of an oxidizing agent. The recovery of uranium from the pregnant leach solution was also studied. Precipitation with alkali yielded a concentrate containing 8% of U_3O_8 .

INTRODUCTION

Very extensive uranium deposits of relatively low grade have been discovered in several localities in northern Saskatchewan such as Charlebois Lake, Black Lake, Freda Lake, Nunn Lake, Foster Lakes, and Lac la Ronge. The uranium in the Charlebois Lake district ores occurs as small crystals of uraninite disseminated in a pegmatitic granite (4). The predominant minerals are feldspar and quartz, but considerable amounts of biotite and smaller amounts of other minerals are also present.

The work to be described was done on portions of a 700 lb. sample of ore that had been collected in the Charlebois Lake region and which was forwarded to the authors by representatives of the Mines Branch in Ottawa. This sample assayed 0.06 to 0.07% U_3O_8 . Extensive leaching tests were carried out and possible methods of recovering the uranium from the pregnant leach solution were studied.

EXPERIMENTAL

Size Reduction

The original rock had been put through a jaw crusher prior to receipt. The particle size was further reduced by two passes through crushing rolls. A sieve analysis of the resulting crushed rock, labelled as sample DE 53-1, is shown in Table I. This table also shows sieve analysis for the same material

¹Manuscript received in original form December 5, 1955, and, as revised, July 16, 1956.

Contribution from the Department of Chemistry, University of Saskatchewan, Saskatoon, Sask., with financial assistance from the Saskatchewan Research Council.

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TABLE I
VARIATION OF SCREEN ANALYSES WITH BALL MILLING TIME,
SAMPLE DE 53-1

Tyler screen No.	Aperture (in.)	Per cent passing screen after ball milling for:						
		0	3	6	8	10	20	45 min.
6	0.1	98	99.3		99.9	100	100	
8	0.093	92.1	97.8	100	99.1	99.5	99.7	
10	0.065	75.2	93.9	98.4	97.4	98.5	99.5	100
14	0.046	53.5	86.5	94.9	94.6	96.8	99.3	99.7
20	0.0328	41.0	79.0	88.8	92.4	95.6	99.1	99.4
28	0.0232	31.5	69.3	82.3	88.7	94.4	98.9	99.1
35	0.0164	23.5	57.3	75.8	78.5	90.7	98.6	98.8
48	0.0116	17.8	41.3	66.3	65.2	82.3	98.2	98.4
65	0.0082	13.3	36.8	55.0	52.3	68.4	96.1	97.8
100	0.0058	9.8	27.7	43.3	40.2	53.0	85.3	96.8
150	0.0041	8.1	23.1	33.3	31.1	40.5	78.5	96.0
200	0.0029	6.2	20.9	26.5	28.0	36.0	63.7	82.9
270	0.0028	4.6					47.7	69.1
400	0.0015	3.0					32.5	44.6

after being subjected to batch ball milling for various times. The charge to the ball mill consisted of 700 gm. of ore; 350 ml. of water; 5 lb. of 0.75 in., 5 lb. of 1 in., and 5 lb. of 1.5 in. nominal diameter steel balls. The 12 in. by 5 in. Denver Laboratory ball mill was rotated at 54 r.p.m. The samples were filtered and dried before screening.

Analysis

Analysis of a representative sample of the ore by standard methods gave the following results:

	Per cent
Ignition loss	1.40
SiO ₂	74.24
Fe ₂ O ₃	5.10
TiO ₂	0.50
Al ₂ O ₃	10.08
CaO	1.20
MgO	1.56
K ₂ O	3.89
Na ₂ O	1.62
U ₃ O ₈	0.07

Spot tests for nickel, cobalt, cerium, and molybdenum were negative. Only trace amounts of carbonate, sulphide, phosphate, and arsenate were present. The low acid-consuming character of this type of ore indicated the attractiveness of an acid leach process.

All uranium analyses were by the fluorophotometric method using equipment similar to that described by Zimmerman (6) except that a Hi Duty Baby Blower, Model 530, was used to circulate air around the mercury vapor lamps to prevent overheating and cracking of the ultraviolet filters. Fusions with 350±20 mgm. of sodium fluoride were done using propane gas and compressed air in a modified Fletcher burner. The fusions were of three minutes duration and steam quenching for one minute was employed.

Uranium analyses of ore samples were carried out as follows. Five-gram samples of finely ground rock were treated with 30 ml. of a 1:1 mixture of

concentrated sulphuric and nitric acids,* evaporated nearly to dryness, taken up in 50 ml. of 1:1 nitric acid, heated nearly to boiling, and made up to a definite volume with water. This treatment did not dissolve the entire sample but experience indicated that satisfactory extraction of the uranium was attained. The samples were filtered prior to subsequent dilution for analysis.

Some difficulty was encountered in obtaining representative samples of the ore for analysis, especially of coarsely ground portions. This difficulty was due to the tendency for the uranium to concentrate in the more finely ground fractions of the ore (3). When a series of leaching tests was to be done, sufficient ore for the whole series was thoroughly mixed before selecting feed samples.

Leaching

Leaching experiments were done in glass-stoppered liter bottles usually charged with 200 gm. of ore and 200 ml. of solution containing specified amounts of reagents. Agitation was achieved by rolling the bottles, in an inclined position, at 35 r.p.m. on a specially constructed rolling table that accommodated 12 bottles simultaneously. All tests were performed at room temperature, $22 \pm 3^\circ\text{C}$.

Samples of pregnant solution were withdrawn at specified intervals with a 1 or 2 ml. pipette, filtered into a volumetric flask, and made up to volume with 1:10 nitric acid. A second dilution was normally required to bring the samples into the concentration range required for fluorophotometric analyses.†

RESULTS AND DISCUSSION OF RESULTS

Leaching Studies

The variables studied were: (1) ball milling time; (2) leaching agent; (3) concentration of leaching agent; (4) leaching time; (5) additives to leaching agents; (6) pulp density. The results are summarized in Tables II to VIII. In all cases the feed ore grade was between 0.06 and 0.07% U_3O_8 . Except as noted the per cent recoveries of U_3O_8 are the mean of at least three fluorophotometric determinations. In some cases averages for several independent leaching experiments are reported in the tables.

From the results reported in Table II it is evident that satisfactory recoveries of uranium (approximately 95%) were obtained by leaching at 50% pulp density with a solution containing 180 lb. of concentrated H_2SO_4 and 56 lb. of concentrated HNO_3 per ton of ore in from 24 to 48 hr., even with comparatively coarse grinds (see Table I for screen analyses). These results also indicate that the recovery of uranium from ore samples ball milled for from 45 to 60 min. is not as high as that obtained with coarser grinds. The lower recoveries with the finer grinds may be due to adsorption of dissolved uranium on colloidal material. Considerably larger quantities of dissolved iron were present in the pregnant solution from the finely ground material. This high iron content was partly due to dissolution of abraded iron from the steel

*All acids referred to in this report were C.P. Concentrated acids of standard commercial strength.

†Dilutions to 0.4×10^{-1} gm. U_3O_8 per 0.1 ml. of solution were considered optimum.

TABLE II

PER CENT RECOVERY OF URANIUM IN SULPHURIC-NITRIC ACID LEACHING OF ORE,
VARIATION WITH BALL MILLING TIME

Weight of ore: 200 gm.

Leach solution: 200 ml. containing 10 ml. conc. H_2SO_4 and 4 ml. conc. HNO_3 ; equivalent to 180 lb. conc. H_2SO_4 and 56 lb. conc. HNO_3 per ton of ore

Normality of original acid leach solution: 2.01

Leaching time (hr.)	Ball milling time (min.)									
	0	3	6	8	10	15	20	32.5	45	60
	Per cent recovery of U_3O_8 in pregnant solution									
7	68									
9		80	85	86	83	78	80	87	77	66
12	80									
24		95	90	96	89	90	81	90	72	72
48					93	92	97	97	83	82
72	90									
Normality of final pregnant solution (phenolphthalein indicator)	1.81	1.77	1.77	1.73	1.79	1.75	1.67	1.65	1.63	1.59

balls used in the grinding. The reducing action of such iron could hardly be responsible for the lower uranium recoveries observed with finely ground ore as nitric acid was present in excess of the iron. The rate at which uranium was dissolved was somewhat greater from the material ball milled for 3 to 15 min. than from the material passed through crushing rolls only. The actual consumption of acid appears to be comparatively small and to increase with increasing ball milling time. The acid normality figures given for the pregnant solution in Table II do not correctly represent actual acid consumption as precipitates of hydrous oxides were formed during the titration.

The data in Table III show the effect of variation in the initial concentration of sulphuric-nitric acid mixtures at 50% pulp density, as well as the effect of ball milling time and duration of leaching. These results show that uranium recoveries of 90% or better were achieved with leach solutions containing as little as 36 lb. of concentrated H_2SO_4 and 11 lb. of concentrated HNO_3 per ton of crushed or coarsely ground material if the leaching time was extended to 72 hr. It is to be noted that the uranium recoveries for the weakest acid solution (E of Table III) at 72 hr. were considerably lower than those obtained at 48 hr. That is, the lower limit of acid concentration for efficient leaching had been exceeded and reprecipitation of uranium salts appears to have occurred. A small per cent loss in weight during leaching was observed. This weight loss was the greater, the higher the acid concentration and the smaller the average particle size of the pulp.

Uranium balances calculated from analyses of the pregnant solutions, washings, and tailings were invariably low when compared to the uranium to be expected from the feed assay. Quenching of fluorescence in the fluoride beads, especially in the case of tailing assays, may be partly responsible for

TABLE III

PER CENT RECOVERY OF URANIUM IN SULPHURIC-NITRIC ACID LEACHING OF ORE OF VARIOUS GRINDS, VARIATION WITH ACID CONCENTRATION

Weight of ore: 200 gm.

Leach solution A: 200 ml. containing 10 ml. conc. $H_2SO_4^a$ and 4 ml. conc. HNO_3^b
 B: 200 ml. " 5 ml. " " " 2 ml. " "
 C: 200 ml. " 3 ml. " " " 1.2 ml. " "
 D: 200 ml. " 2 ml. " " " 0.8 ml. " "
 E: 200 ml. " 1.25 ml. " " " 0.5 ml. " "

Leaching time (hr.)	Leach solution	Ball milling time (min.)				
		0	3	6	10	20
		Per cent recovery of U_3O_8 in pregnant solution				
12	A	80	84 ^c	87 ^c	85 ^c	80 ^c
	B	89			80	85
	C	78	82	83	83	85
	D	75	72	79	83	83
	E	61	72	75	77	77
24	A	85 ^c	95	90	89	81
	B	90			87 ^c	88 ^c
	C	82	85	85	81	85
	D	76	78	82	85	81
	E	70	73	78	78	82
48	A	89 ^c			93	97
	B	93			98	94
	C	89	93	90	88	87
	D	83	84	85	86	85
	E	77	87	83	80	82
72	A	90				
	C		88	86	90	92
	D	97	91	88	87	87
	E		69	67	65	69
Per cent U_3O_8 in dried tailings	B				1.7	2.5
	C		3		3	3
	D		5	5	5	5
Loss in weight of ore during leaching period (gm.)	B				9	14
	C		4.6	4.9	5.0	6.9
	D		1.8	3.0	4.5	5.8

^a1 ml. of conc. H_2SO_4 corresponds to 18 lb. of conc. acid per ton of ore.^b1 ml. of conc. HNO_3 corresponds to 14 lb. of conc. acid per ton of ore.^cValue interpolated from leaching results for other times.

this effect. Uranium recoveries reported in this paper are based on the feed assay.

The effect of varying the pulp density while maintaining a constant ratio between the amount of acid and the amount of ore is illustrated in Table IV. Although differences in the leaching efficiency were not marked, that for the 40% pulp density was definitely lower than that for the 50 and 67% pulp densities. This effect may be due to dilution of the acid. The differences in the recoveries of uranium between the 50 and 67% pulp densities were not sufficiently great to justify drawing any definite conclusion. The difficulty of mixing puts an upper limit on the pulp density that can be effectively employed.

TABLE IV

PER CENT RECOVERY OF URANIUM IN SULPHURIC-NITRIC ACID LEACHING OF CRUSHED ORE,
VARIATION WITH PULP DENSITY (ACID CONCENTRATION)

Weight of crushed ore (<0.185 in.): 200 gm.

Leach solution contained 2 ml. conc. H_2SO_4 and 0.8 ml. conc. HNO_3 in each case

Leaching time (hr.)	Total volume of leach solution (ml.)		
	300	200	100
	Approximate pulp density (per cent solids by weight)		
	40	50	67
	Per cent recovery of U_3O_8 in pregnant solution		
4	53	68	73
8	66	77	85
24	78	—	—
48	80	83	85
72	86	97	95

Table V is a summary of results obtained using various concentrations of H_2SO_4 alone as the leaching agent on the crushed rock. In general, uranium recoveries were observed to decrease for the same period of leaching as the amount of sulphuric acid was reduced from 72 lb. to 22.5 lb. per ton of crushed ore. A uranium recovery of 80% was obtained in 48 hr. by using as little as

TABLE V

PER CENT RECOVERY OF URANIUM IN SULPHURIC ACID LEACHING OF ORE,
VARIATION OF CONCENTRATION OF SULPHURIC ACID

Weight of crushed ore (<0.185 in.): 200 gm.

Leach solution: 200 ml. containing conc. H_2SO_4 as indicated

Leaching time (hr.)	Volume of conc. H_2SO_4 added (ml.)			
	4	3	2	1.25
Per cent recovery of U_3O_8 in pregnant solution				
2	44	37	41	43
6	64	67	60	46
12	73	80	67	65
24	77	80	77	69
48	88	90	80	74
pH of original leach solution	0.5	0.8	0.9	1.2
pH of final pregnant solution	1.4	1.6	2.0	2.8

36 lb. of concentrated H_2SO_4 per ton of ore. The same recovery was obtained in 24 hr. using 54 lb. of concentrated H_2SO_4 per ton of crushed ore. The pH changes noted in Table V indicate that a large fraction of the acid added was consumed in the leaching process.

The effect of adding HNO_3 , $KClO_3$, HCl , KCl , and K_2SO_4 to a leach solution containing 36 lb. of concentrated H_2SO_4 per ton of crushed ore is illustrated in Table VI. The addition of 11.2 lb. of concentrated HNO_3 , 8 lb. of $KClO_3$, 8 lb. of KCl , or 8 lb. of K_2SO_4 per ton of ore did not significantly change the

TABLE VI

PER CENT RECOVERY OF URANIUM IN SULPHURIC ACID LEACHING OF ORE,
EFFECT OF VARIOUS ADDITIVES

Weight of crushed ore (<0.185 in.): 200 gm.

Leach solution: 200 ml. containing 2 ml. of conc. H_2SO_4 and other reagents as indicated

Leaching time (hr.)	Additive							
	None	KCl, 2 gm.	KCl, 0.8 gm.	Conc. HCl, 1 ml.	Conc. HCl, 0.8 ml.	K_2SO_4 , 0.8 gm.	$KClO_3$, 0.8 gm.	Conc. HNO_3 , 0.8 ml.
	Per cent recovery of U_3O_8 in pregnant solution							
2	41	37	39	41	45	41	50	47
6	60	56	37	57	63	60	65	61
12	67	77	70	77	81	72	70	75
24	77	87	69	83	81	69	76	76
48	80	96	79	91	87	80	83	78

TABLE VII

PER CENT RECOVERY OF URANIUM IN HYDROCHLORIC ACID LEACHING OF ORE,
VARIATION WITH CONCENTRATION OF HYDROCHLORIC ACID

Weight of crushed ore (<0.185 in.): 200 gm.

Leach solution: 200 ml. containing conc. HCl as indicated

Leaching time (hr.)	Volume of conc. HCl added (ml.)		
	4	3	2
	Per cent recovery of U_3O_8 in pregnant solution		
2	37	33	29
6	43	39	33
12	46	40	34
24	51	44	34
48	53	42	31
pH of original leach solution	0.8	1.0	1.3
pH of final pregnant solution	2.1	2.6	2.9

TABLE VIII

PER CENT RECOVERY OF URANIUM IN SULPHURIC ACID LEACHING OF ORE,
EFFECT OF VARIOUS ADDITIVES

Weight of ore: 200 gm.

Ball milling time: 3 min.

Leach solution: 200 ml. containing 2 ml. conc. H_2SO_4 and additives as indicated

Leaching time (hr.)	Additive				
	0.8 ml. conc. HNO_3	2 ml. 30% H_2O_2	2 gm. $Na_2S_2O_8$	2 gm. $KMnO_4$	2 gm. $Na_2Cr_2O_7$
	Per cent recovery of U_3O_8 in pregnant solution				
4	68	71	75	74	71
8	76	75	79	82	73
24	78	78	81	84	85
48	84	91	88	—	—
72	91	92	92	91	88

leaching efficiency of the sulphuric acid. However, the addition of 20 lb. of KCl or 11.5 to 9.2 lb. of concentrated HCl to the sulphuric acid did result in an increase in the uranium recovery after 24 hr. of leaching. In contrast, hydrochloric acid alone proved rather ineffective as a leaching agent as is illustrated by the results shown in Table VII.

Table VIII shows the effect of various other additives on sulphuric acid leaching of the ore. If the results in Tables V and VIII are compared, it appears that there is little advantage in using additives such as concentrated HNO_3 , 30% H_2O_2 , $\text{Na}_2\text{S}_2\text{O}_8$, KMnO_4 , or $\text{K}_2\text{Cr}_2\text{O}_7$ in sulphuric acid leach solutions containing 36 lb. of concentrated H_2SO_4 per ton of ore. The rate of leaching was increased by some of these additives but the final uranium recovery after 48 to 72 hr. of leaching was not found to be appreciably different from that obtained with sulphuric acid alone.

In view of the established commercial practice of using chlorates or other oxidizing agents as aids in acid leaching of uranium ores, these results indicate that the concentration of ferric ions in the leach liquor must be sufficient to effectively oxidize uranium IV to uranium VI. The results of typical analyses of pregnant liquors obtained by leaching crushed ore for 48 hr. with a solution containing 36 lb. of conc. H_2SO_4 per ton of ore are shown in Table IX. The

TABLE IX

SULPHURIC ACID LEACHING OF ORE

Crushed ore (<0.185 in.) leached for 48 hr. at 50% pulp density with a solution containing 36 lb. of conc. H_2SO_4 per ton of ore

	Sample 1	Sample 2
Feed assay (% U_3O_8)	0.055	0.067
Tailing assay (% U_3O_8)	0.0074	0.0088
Per cent of original solids dissolved	2.0	2.0
Per cent recovery of U_3O_8 in pregnant solution and washings*	86.8	87.1
pH of original acid	0.90	0.90
pH of pregnant solution	1.60	1.63
U_3O_8 in pregnant solution (gm./liter)	0.480	0.544
Fe^{+++} in pregnant solution (gm./liter)	1.6	1.7
Fe^{+++} in pregnant solution (gm./liter)	0.4	0.8
SO_4^{--} in pregnant solution (gm./liter)	16.4	16.3

*Tailings were repulped and washed twice with dilute H_2SO_4 solution (pH 1.78).

concentration of ferric ion in the pregnant solution is sufficient to maintain a potential of approximately -400 mv. The presence of ferric iron accounts for the successful leaching of this ore without the addition of an oxidizing agent. Other than iron, the only contaminants found to be present in the pregnant solution from sulphuric acid leaching were aluminum, calcium, magnesium, potassium, sodium, and silica.

It was concluded that uranium recoveries of 90% or better could be obtained by leaching the pegmatitic ore at 50 to 60% pulp density for periods of from 48 to 72 hr. with a solution containing 36 lb. of concentrated H_2SO_4 per ton

of crushed ore (<0.185 in. 23% minus 35 mesh). The leaching period can be shortened by increasing the initial acid concentration or by reducing the average particle size of the ore to 50 to 75% minus 35 mesh. However, overgrinding is to be avoided as it results in a decreased uranium recovery and an increased concentration of contaminants in the pregnant solution. In plant practice an economic balance would have to be made between grinding costs and the cost of increased leaching tank space.

Since sulphuric acid is the essential chemical required for the successful leaching of this pegmatitic ore and the actual acid consumption is relatively small, comparatively low cost leaching is possible. The practicability of sulphuric acid leaching of these ores would be increased if workable deposits of pyrite, or other sulphide ores which could be used for acid manufacture, were discovered in the immediate vicinity of the uranium mine.

In plant practice leaching efficiency could probably be increased by using a countercurrent flow process. The possibility of using a heap method should also be considered, although it would have the disadvantage of being practical only in the summer season. Depending on the cost of sulphuric acid, it might be possible to economically leach pegmatitic ores containing 0.06 to 0.07% of U_3O_8 by spraying acid solution over heaps of crushed ore piled on an impervious base provided with adequate drainage to facilitate collection of the pregnant solution. Recirculation of the pregnant solution through the heap could be carried on until the acid concentration became too low for efficient leaching. Leaching of the ore heap would be continued until the uranium recovery became unprofitable. The economics of this or other possible leaching methods still remain to be considered.

Recovery of Uranium from Pregnant Solutions

The pregnant solutions from sulphuric acid leaching contained about 0.6 gm./liter of U_3O_8 together with 2-3 gm./liter of iron as the principal contaminant. Some preliminary tests of chemical precipitation methods for recovery of uranium from these solutions were carried out. The aluminum reduction - phosphate precipitation method (2, 5) gave satisfactory uranium recoveries, but was not considered practical in that an additional 20 lb. of conc. H_2SO_4 per ton of leach solution was required to lower the pH to the optimum value for phosphate precipitation. Complete precipitation of uranium from unreduced pregnant solutions of pH 1.6 required the addition of from 16 to 20 lb. of concentrated ammonia or NaOH per ton of solution. From 13 to 15 lb. of alkali was required when the pH of the pregnant solution was approximately 2.0.

The dried concentrate obtained by precipitation of the uranium values from pregnant solutions with alkali contained the equivalent of 8% U_3O_8 , representing a somewhat greater than one hundred fold increase in the uranium content over that in the original ore. This does not compare favorably with concentrates that can be obtained by ion exchange methods (1). Further work on both ion exchange and solvent extraction methods of recovering uranium from pregnant solutions is contemplated.

ACKNOWLEDGMENT

The assistance of E. C. Bailey and L. W. Crawford in carrying out certain phases of this work is acknowledged.

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DESCRIPTION OF 5-LITER STAINLESS STEEL FERMENTORS¹

BY J. M. ROXBURGH, J. F. T. SPENCER, AND H. R. SALLANS

ABSTRACT

Five-liter stainless steel laboratory fermentors of particularly simple and rugged design are described. These have given reliable and reproducible results in the investigation of fermentation variables. Yields of ustilagic acid from *Ustilago zeae* fermentations are given to indicate the performance obtainable from them. Oxygen absorption coefficients have been determined for these and larger fermentors and used successfully to scale up the production of ustilagic acid.

INTRODUCTION

Small scale equipment is almost essential if sufficient replication for the successful investigation of environmental and nutritional factors influencing fermentation processes is to be obtained. However, if the most value is to be obtained from the data, it must be possible to use them to predict desirable design and operating conditions for pilot plant and production scale fermentors. If aerobic fermentations are to be scaled up, a method must be found for correlating agitator speeds and air flow rates.

The correlation determined from the rate of oxidation of aqueous sodium sulphite solutions catalyzed by copper ion (3) has been applied to penicillin and streptomycin fermentations (5) and to the production of ustilagic acid (6). The method is relatively simple but is indirect and cannot be considered to be universally applicable. Shu (7) has indicated at least three limitations. Fermentations which require high oxygen tension during growth, which do not show a direct relation between oxygen uptake and product formation, or which undergo marked changes in physical properties during the course of the fermentation may require the application of direct methods of determining available oxygen in the broth (2, 4, 7, 8, 10).

If these limitations are kept in mind, the sulphite method seems preferable for those fermentations for which it is adequate. Oxygen absorption coefficients can be determined directly or they can be estimated from the correlation given by Cooper, Fernstrom, and Miller (3), though some method of measuring the power input to the agitator is required in the latter case. Since the water solution of sulphite normally used in the direct measurement of oxidation rates differs in viscosity and other physical characteristics from fermentation media, the results obtained are not an exact measure of the conditions actually occurring in a fermentation. A better measurement for any particular fermentation can be obtained by using fermentation media plus the killed cells or mycelium instead of water, but this is seldom practical except in small scale equipment.

¹Manuscript received June 4, 1956.

Contribution from the National Research Council of Canada, Prairie Regional Laboratory, Saskatoon, Saskatchewan. Issued as Paper No. 229 on the Uses of Plant Products and as N.R.C. No. 4088.

In these laboratories, 200-gal. (U.S.) stainless steel fermentors were installed for pilot plant operations. The small fermentors described in this paper (Fig. 1) were designed to obtain data in the preliminary stages of the

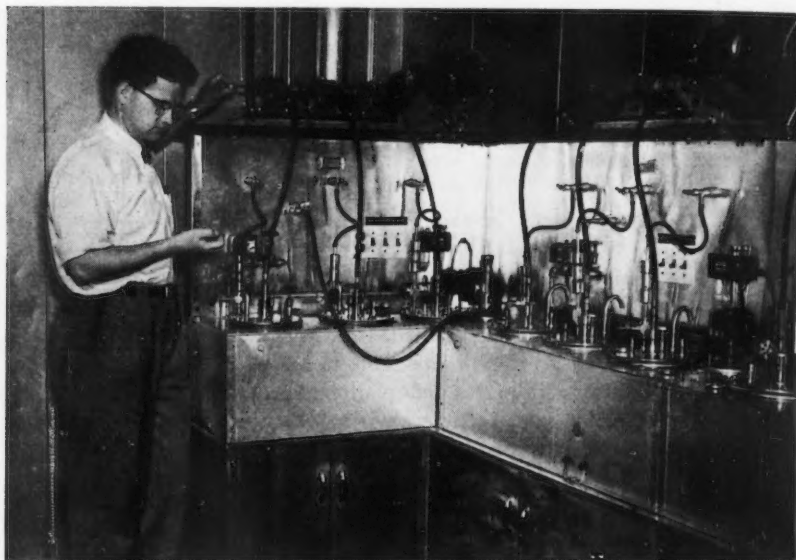


FIG. 1. Five-liter fermentors.

development of new fermentations adequate for the prediction of suitable operating conditions in the large fermentors. To this end sulphite oxidation rates were measured over the full range of operating conditions for both sizes of fermentors. Twenty-liter fermentors are also used, primarily for the production of inoculum for the 200-gal. size, and their characteristics were also determined. Because of high losses in the agitator drives in the large fermentors measurements of power input were not attempted.

EXPERIMENTAL

Eighteen 5-liter fermentors were made, using straight sided, lipless, stainless steel beakers of 5800 ml. capacity for the bodies, and with all fittings attached to the cover.

The arrangement of the parts is shown in Fig. 2. Stainless steel was used throughout, and fittings are all of readily available standard sizes. The cover proper is a $9\frac{1}{2}$ in. circle of 12 gauge ($\frac{1}{8}$ in. approximately) plate. The supports for the three baffles (3) are $\frac{1}{8}$ in. pipe with $\frac{1}{4}$ in. half nipples (8) reamed to fit and welded to them. These are threaded into the lid so that the baffles are in proper position when tightened into place. One baffle support is bent at right angles, capped, and a $\frac{3}{64}$ in. hole (1) drilled directly below the agitator to

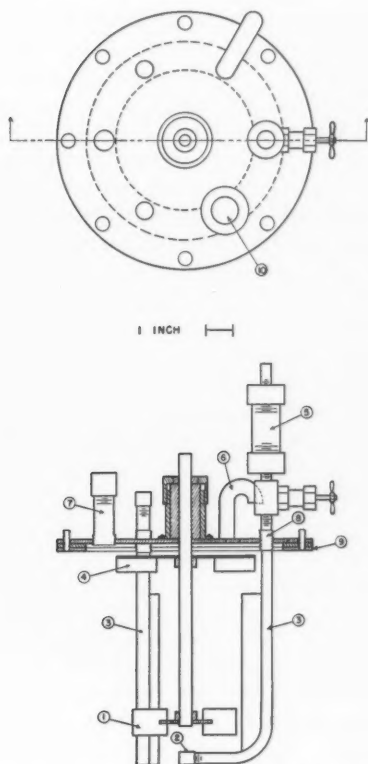


FIG. 2. Five-liter fermentor. Details of construction for the lid and agitator assembly.

- | | |
|--------------------|---|
| 1. Impeller | 6. Air outlet |
| 2. Air sparger | 7. Inoculum and sample tube |
| 3. Baffle supports | 8. Half nipple welded to baffle support |
| 4. Foam breaker | 9. Clamping ring and gasket |
| 5. Air filter | 10. Sight glass |

serve as an air inlet. The other two supports are normally capped, and their bottom ends are welded closed, but for special purposes (e.g. nutrient addition during fermentation) they may be replaced by suitable modifications. The baffles are 14 gauge stainless steel strips welded to the supports. The agitator disk and hub are preferably turned from solid stock (2 in. diam.) but earlier models were made by welding a hub ($\frac{3}{4}$ in. rod) to a 14 gauge disk. The agitator blades (2) are 1 in. by $1\frac{1}{2}$ in. of 14 gauge stainless steel, slotted and welded to the disk. The hub is drilled to fit the $\frac{3}{8}$ in. diam. shaft and secured with two set screws. The foam breaker (4) is similar but of lighter construction. Disk and blades are of 18 gauge stainless steel spot-welded or bolted together and welded to the $\frac{3}{4}$ in. diam. hub. The shaft runs in an "Oilite" bearing mounted on the lid as shown, and is held in position by a collar secured with set screws.

The air outlet (6) and sampling tube (7) are of stainless steel pipe threaded to the lid. An air filter and inlet air valve (5) and a sight glass (10) are also fitted. The agitator and cover assembly are held securely to the beaker rim by the clamping ring (9) and eight thumb screws. A rubber gasket between the lid and the beaker makes an adequate seal.

The fermentors are mounted in controlled temperature water baths in groups of three, each group having a common stirrer drive through three gear reducers and flexible shafts (Fig. 2). Agitator speeds from 200 to 900 r.p.m. are obtained by using a variable speed motor pulley with two sizes of fixed pulleys on the gear reducer shaft. Thus 18 fermentations can be run simultaneously with up to six different agitator speeds and temperatures, and with all other variables individually controlled in each fermentor.

The design provides a maximum of flexibility. Impellers, baffles, and foam breakers are readily altered or replaced to suit conditions desired for a specific fermentation. Straight blade turbine type impellers have been used in the standard design since they are most easily constructed and adjusted to give replication between fermentors. Arrowhead impellers, used to give high aeration levels at relatively low speeds in some earlier designs, were difficult to adjust to give uniform conditions in all fermentors.

Aeration efficiencies of these fermentors were determined over a range of impeller speeds and air flow rates for two sizes of impellers. Three liters of 5% sodium sulphite solution was placed in the fermentor at $25^{\circ} \pm 0.5^{\circ}\text{C}$. and 20 ml. samples were taken at 5 or 10 min. intervals without stopping the agitation or flow of air. Under these conditions the rate of oxygen uptake is linear with time and can be taken as a measure of the oxygen absorption coefficient (3). The samples were added to an excess of standard iodine solution and back-titrated with standard sodium thiosulphate.

Fig. 3 shows typical results for these fermentors at a constant air flow rate of 1 liter per minute. The large impeller is as described previously and the

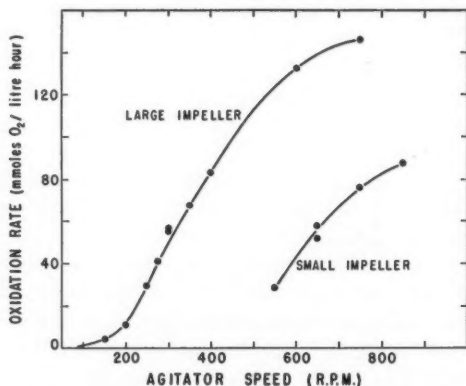


FIG. 3. Effect of agitator speed on the oxygen absorption coefficient at a constant air flow rate of 1 liter per minute in 5-liter fermentors. Superficial air velocity 7.91 ft. per hr.

small one was of similar construction but $2\frac{3}{4}$ in. in over-all diameter with blades $\frac{1}{2}$ in. by $\frac{3}{4}$ in. All fermentors were adjusted (by moving the baffles and impeller position) to give the same rate of sulphite oxidation at 300 r.p.m. and with 1 liter of air per minute. Over the range of 300 to 600 r.p.m. all fermentors gave rates within 4% of the mean value. Since the air flow rate was not measured with greater accuracy than this the results were accepted as indicating satisfactory agreement.

The significant factors affecting the efficiency of aeration at constant agitator speed and air flow rate were found to be the distance from the air sparger to the bottom of the impeller and the angle between the baffles and the tangent to the fermentor wall. As the impeller is moved farther from the air inlet a progressive and nearly linear reduction in aeration efficiency results, amounting to about 10% per inch under normal conditions of air flow rate and agitator speed. The baffles operate most efficiently when at right angles to the wall of the fermentor, with a reduction of about 5% in aeration efficiency for a five degree deflection from the normal.

The effect of air flow rate on the oxygen absorption coefficient is shown in Fig. 4 for the larger impeller size. Data were obtained on all fermentors and

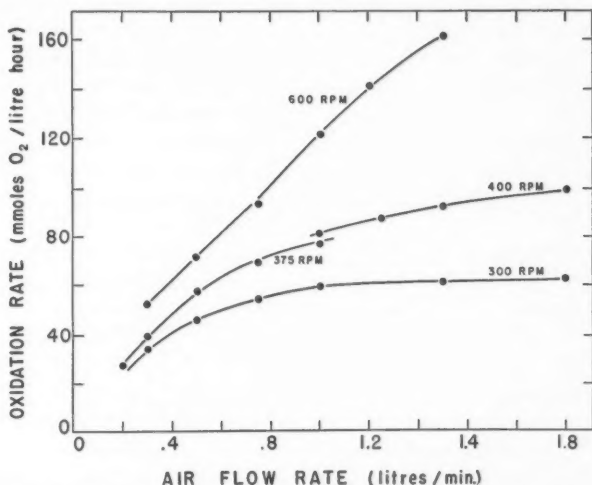


FIG. 4. Effect of air flow rate on the oxygen absorption coefficient in 5-liter fermentors with the $3\frac{1}{2}$ in. diam. impeller. Superficial air velocity in ft. per hr. is 7.91 times air flow rate shown.

the average values plotted. Standard deviations from the mean decreased from a maximum of 6% at low air flow rates to 3.5% at higher flows.

The range of aeration conditions available in the three sizes of fermentors is shown in Table I. The maximum sulphite oxidation rate for the 200-gal. fermentor is approximately two thirds of that for the other two sizes and is attained only at very high superficial air velocities. Higher agitator speeds

TABLE I
TYPICAL SULPHITE OXIDATION RATES FOR 200-GAL, 20-LITER, AND 5-LITER FERMENTORS

Fermentor	Superficial air velocity, ft./hr.	Agitator speed, r.p.m.	Sulphite oxidation rate ^a mM. O ₂ /liter/hr.
200-gal. ^b	70.7	200	93
	47.2	150	48
	23.6	100	14
20-liter	28.8	400	143
	19.2	345	70
	14.4	225	32
5-liter ^c	11.86	600	158
	7.91	200	16

^aAt 1 atm. pressure.

^bImpeller 13 in. O.D.

^cImpeller 3½ in. O.D.

cannot be obtained with the power available. Because of excessive foaming, it is seldom possible to run fermentations under conditions equivalent to those producing the highest oxidation rates shown, even with the aid of antifoam agents and mechanical foam breakers.

Yields of ustilagic acid produced by *Ustilago zeae* in submerged culture (9) were used as a measure of the degree of replication obtainable with the 5-liter fermentors. This fermentation is affected considerably by the aeration level maintained during growth, both in rate and yield (6). Table II shows yields from 12 fermentations run at the same time on the same medium from pooled inoculum. The respective rates of utilization of dextrose were the same for all

TABLE II
REPRODUCIBILITY OF *U. zeae* FERMENTATIONS IN 5-LITER FERMENTORS. SULPHITE OXIDATION RATE UNDER EQUIVALENT CONDITIONS, 82.0 MILLIMOLES OF OXYGEN PER LITER PER HOUR

Fermentor No.	Yield of ustilagic acid, gm./liter	Average yield for bank of three fermentors
1	11.6	10.9
2	10.6	
3	11.7	
4	13.2	12.5
5	11.5	
6	12.7	
7	10.8	10.8
8	11.6	
9	10.1	
10	11.0	11.1
11	11.0	
12	11.4	
Average	11.4 S.D. ± 0.86 ($\pm 7.5\%$)	

fermentations in the set within the accuracy of the determination. Yields from fermentations run at different times showed standard deviation of 8%.

Table III shows yields of ustilagic acid and comparable sulphite oxidation rates in shake flasks, 5-liter fermentors, and a 200-gal. pilot plant fermentor. Although widely different superficial air velocities were used to obtain the

TABLE III

SULPHITE OXIDATION RATES AND YIELDS OF USTILAGIC ACID IN FERMENTORS AND SHAKE FLASKS

Fermentation unit	Agitator speed, r.p.m.	Superficial air velocity, ft./hr.	Sulphite oxidation rate, mM. O ₂ /liter/hr.	Yield of ustilagic acid, gm./liter
200-gal. ^a pilot plant	400	47.2	76.8	15.0
5-liter laboratory	375	7.91	77.0	15.8 ^c
500-ml. flasks, 50-ml. medium	225 ^b	—	18.0	14.3 ^c

^aImpeller 8 in. O.D.

^bRotary shaker, eccentricity 1 in.

^cAverage of three.

same apparent aeration in the two sizes of fermentor, yields compare very well. The data from shake flasks indicates clearly that a certain minimum degree of geometric similarity is necessary between fermentation vessels for the successful application of this method. The higher yields at lower aeration levels shown here as compared with Table II resulted from the use of different strains of the organism.

DISCUSSION

The 5-liter fermentors described in this paper have been useful in the investigation of fermentation variables in these laboratories. The *U. zeae* fermentation has been studied in detail (6) and other fermentations are currently being investigated. While these fermentors do not differ in principle from some of those described by other workers (e.g. (1, 5)) they have the advantages of simplicity of construction and extreme ruggedness, the latter characteristic eliminating entirely losses due to breakage during handling and operation. They have, as well, been found to give dependable and reproducible performance.

Substantially the same design, with the addition of internal cooling coils, has been used in the construction of "Pyrex" fermentors where complete visibility was required. These are satisfactory in most respects but are fragile, and temperature cannot be controlled as easily or as precisely as in the stainless steel model.

The sulphite oxidation method used to correlate aeration conditions between the various sizes of fermentors used is standard. The differences in design between our smaller fermentors and the 200-gal. ones are appreciable, particularly in respect to the power to volume ratios available, but they are

apparently not great enough to prevent the application of this comparatively simple scale-up method to the *U. zeeae* fermentation and, presumably, to other fermentations with similar favorable characteristics.

ACKNOWLEDGMENT

The authors wish to acknowledge the technical assistance of M. Anderson and A. E. York in the construction of these fermentors.

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NOTE

A SIMPLE APPARATUS FOR THE CHANGE-OVER FROM REFLUX TO DISTILLATION¹

By H. F. BAUER

It is common practice in the organic chemistry laboratory to carry out a reaction under reflux and then to remove one or more of the components by distillation. The change from a reflux to a descending distillation condenser, however, is tedious and time consuming. Also, depending on the skill of the operator the contents of the flask is exposed for a shorter or longer period to the surrounding atmosphere; when moisture, carbon dioxide, or oxygen has to be excluded from the reaction, this is highly undesirable.

These difficulties are readily eliminated by use of the apparatus shown in Fig. 1. The standard taper joints are connected with a condenser and the

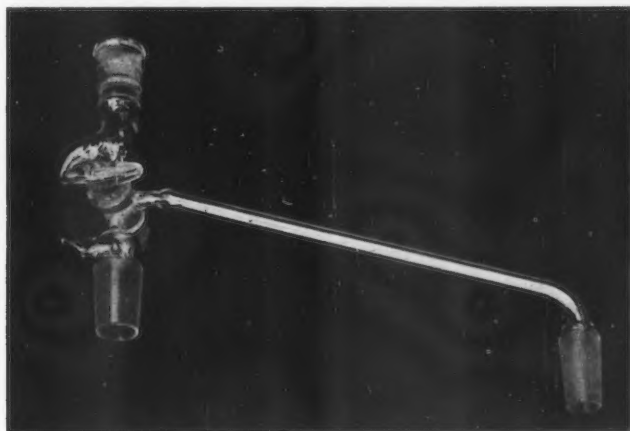


FIG. 1.

distilling and receiving flasks, and the change from boiling under reflux to the distillation is carried out merely by turning the "T" three-way stopcock. To facilitate the escape of air trapped in the receiving flask, a groove is ground into the stopcock connecting the two bores of the "T" which in the distilling position guide the stream of distillate into the receiver. The danger of breakage, although very small on the model shown, can be further decreased by the use of ball joints. Separate fractions can be collected easily by temporarily switching the stopcock to the reflux position and changing the receiver. Vacuum

¹Issued as N.R.C. No. 4094.

distillations are carried out by applying the vacuum at the top of the condenser; the shape of the distilling head is such that splashing of liquid into the receiver is avoided. The stopcock is held in position by a spring and if well ground is lubricated by the distillate and does not require additional lubricant; if grease must be applied, a nonsoluble one can easily be selected from the wide variety available today.

The author has used the described apparatus in his own laboratory for several years with great advantage. It has proved particularly useful for the purification and drying of solvents.

RECEIVED JULY 16, 1956.
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